2.1.1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
OREGON OPERATIONS OFFICE
811 S.W. 6th Avenue
Portland, Oregon 97204

RECEIVED
MAY 18 2004
Environmental Cleanup Office

May 12, 2004

Mr. Jim McKenna
Port of Portland & Co-Chairman, Lower Willamette Group
121 NW Everett
Portland, Oregon 97209

Mr. Robert Wyatt
Northwest Natural & Co-Chairman, Lower Willamette Group
220 Northwest Second Avenue
Portland, Oregon 97209

Re: Portland Harbor Superfund Site; Administrative Order on Consent for Remedial Investigation and Feasibility Study; Docket No. CERCLA-10-2001-0240
Round 2 Quality Assurance Project Plan

Dear Messrs. Wyatt and McKenna:

We have completed our review of the April 12, 2004 draft Round 2 Quality Assurance Project Plan (QAPP). There are a number of revisions needed prior to EPA approval of this document and in response to EPA's conditional approval of the Round 2 Field Sampling Plan for Sediment Sampling and Benthic Toxicity Testing. In addition some revisions will be needed in relation to our forthcoming comments on the Round 2A Surface Water Field Sampling Plan. These relate primarily to sediment sample numbers, surface water sample numbers, analyses and locations, and the 20-Day Chironomus bioassay test.

EPA's comments are attached. Please submit a revised Round 2 QAPP that addresses the attached comments within 30 days following the date of this letter.

Please contact Chip Humphrey at (503) 326-2678 or Eric Blischke (503) 326-4006 to set up this meeting or if you have any questions. All legal inquiries should be directed to Lori Cora at (206) 553-1115.

Sincerely,

Chip Humphrey

Chip Humphrey
Eric Blischke
Remedial Project Managers

cc: John Crellin, ATSDR
Helen Hillman, NOAA
Ted Buerger, US Fish and Wildlife Service
Preston Sleeger, Department of Interior
Jim Anderson, DEQ
Kurt Burkholder, Oregon DOJ
Rick Keppler, Oregon Department of Fish and Wildlife
David Stone, Oregon Public Health Branch
Rod Thompson, Confederated Tribes of Grand Ronde
Tom Downey, Confederated Tribes of Siletz
Audie Huber, Confederated Tribes of Umatilla
Brian Cunninghame, Confederated Tribes of Warm Springs
Rick Eichstaedt, Nez Perce Tribe
Paul Ward, Confederated Tribes of Yakama Nation
Valerie Lee, Environment International
Patti Howard, CRITFC
Keith Pine, Integral Consultants

General Comments:

Scope of QAPP

The Round 2 Quality Assurance Project Plan (QAPP) covers surface water, sediment chemistry, and sediment toxicity bioassays. However, other sampling elements (groundwater and natural attenuation) are contemplated during Round 2. It is our expectation that QAPP revisions will be completed for the other sampling elements, either as addendums to the QAPP or as stand -alone documents. We will be discussing QAPP requirements for these Field Sampling Plans (FSPs) with the LWG in the near future.

Round 2 Surface Water:

At this time, the EPA and its partners are still reviewing the Round 2 Surface Water FSP. In addition, key elements of the Round 2 QAPP related to surface water have not been provided pending selection of a surface water laboratory. These include laboratory methods, method detection limits (MDLs), method reporting limits (MRLs) and the surface water laboratory Quality Assurance Manual. EPA and its partners will be commenting on a number of elements related to the Round 2 QAPP in its comments on the Round 2 Surface Water FSP. It is EPA's expectation that our comments on the Round 2 Surface Water FSP will be incorporated into the revised QAPP.

The LWG accepted EPA's direction to include low detection limit methods and alternative concentration goals (ACGs) for surface water. The ACGs that have been included in the QAPP are default values that are not based on site-specific parameters that have been discussed between LWG, EPA and agency partners. In particular, the human health screening values, upon which many of the low detection ACGs were based, were calculated using generic fish consumption values. The QAPP should note that higher fish consumption rates have been agreed to in the programmatic work plan and that site specific data generated and food web modeling may require adjustment of ACGs in subsequent surface water sampling efforts.

Sample Numbers:

The sample count presented in the QAPP appears low. The numbers should be recalculated in accordance with the conditionally approved Round 2 Sediment Sampling and Benthic Toxicity Testing (Sediment and Bioassay) FSP and EPA comments on the Round 2 Surface Water FSP. This includes surface sediment, subsurface sediment and surface water samples. The QAPP should include Tables 2-2 and 2-3 from the Round 2 Sediment and Bioassay FSP and applicable tables that depict sample numbers, locations and analyses from the Round 2 Surface Water FSP.

20-Day Chironomus Bioassay Test:

At this time, EPA has directed the LWG to perform the 20-day Chironomous test instead of the

10-day test. The 20-day test must be incorporated into the revised QAPP.

Data Validation:

Except for the Columbia Analytical Services (CAS) laboratory in Kelso, Washington, the performance and capabilities of the other the designated laboratories are unknown. Since Round 2 will be a significant data collection effort, the first data package generated by each lab for each suite of parameters should be submitted to EPA for evaluation and data quality assessment. By doing this, any problems with the analytical methods and or laboratory performance can be identified during the early phase of the project.

Specific Comments:

Section A3 - Distribution List: Please correct the following typographical errors:

- "Yakama" Nation
- remove "DNR" before "Confederated Tribes of the Umatilla Indian Reservation" or move it after "Reservation" separated by a comma
- "Helen" Hillman

Figure A4-1 Round 2 Project Organization: The figure should reference all Round 2 sampling activities and their laboratory analysis coordination.

Section A4.2.1 - EPA Organization and Responsibilities: Rene Fuentes (hydrogeologist) should be added as an EPA staff person with significant involvement in the Portland Harbor RI/FS and in Table A4-1 under EPA Region 10.

Section A5.1 - Portland Harbor RI/FS: In the second line on page 6 after "contact with sediments", add "surface water and seeps."

Section A5.2 - Round 2 Sampling:

This section should describe the relationship of the QAPP to the various Round 2 data collection efforts including groundwater sampling, natural attenuation sampling and Round 2A and 2B sediment coring.

In the first bullet of this section add the words "surface and" before "buried sources". In the first bullet of the next section of bullets, add "and human health" after "support the ecological."

Section A6.1.1 - Surface and Subsurface River Sediment: The referenced tables (Tables 2-2 and 2-3 of the Round 2 Field Sampling Plan) should be included in the QAPP.

Section A6.1.2 - Beach Sediment: The LWG should analyze beach sediment samples in human use areas in accordance with the Attachment B of EPA's April 23, 2004 comments on the Round

2 Field Sampling Plan - "Explanation regarding use of Total Petroleum Hydrocarbon Data."

Section A6.1.3 - Surface Water: The QAPP does not adequately state the project objectives for surface water and how these objectives will be achieved. This section must be revised in accordance with EPA comments on the Round 2 Surface Water FSP.

Section A6.2 - Laboratory Analyses and Deliverables: Please add the following statement to paragraph 3: "A list of hard copy data deliverables from the laboratory is discussed in detail in section A9.2 of the QAPP."

Section A6.3 - Data Quality Evaluation: This section should clarify the laboratory data verification procedures. It should state who is responsible for performing the verification (e.g., laboratory analyst, laboratory QA officer). In addition, it is more appropriate to verify the analytical results based on the technical specifications of the QAPP and the method requirements and not on the lab's SOPs or QA Manual. At the top of page 12, replace the "secondary" data verification to "third party" data verification.

Section A6.6 - Project Schedule:

The QAPP states that laboratory data will be due to LWG 30 days from the receipt of the last sample in each sample batch. The QAPP should define what constitutes a sample batch.

Section A7.2 - Data Quality Indicators:

In the second paragraph on page 15, change "requested" to "generated" data per suite of parameters per sampling event.

The Measurement Quality Objectives listed in Tables A7-1 and A7-2 must be used by the laboratories to determine if re-analysis are warranted or not. A copy of the EPA approved Round 2 QAPP or, at a minimum, Tables A7-1 and A7-2 should be provided by LWG to the laboratories.

Add the following bullets to Page 15: (1) "standard verification" and (2) " holding blanks" for VOCs and the suite of parameters requiring ultra low level detection limits.

The second sentence of the second paragraph on page 16 should be revised to read: The methods and modifications selected for this study will incorporate modifications recommended by PSEP (1997a) or the specified analytical methods to optimize MRLs.

The discussions regarding MDLs on pages 15 and 16 should be moved to Section B4, Analytical Methods of the QAPP.

Section A9 - Documents and Records: This section should specify who will maintain the

documentation relating to sample collection, laboratory analysis and bioassays. Three types of data will be generated at the lab: (1) instrument and raw data output stored in tapes/cartridges; (2) electronic data deliverables; and (3) the reduced and verified data stored in the LIMS. These data should be archived by the laboratory and the length of time the laboratory is required to store them should be stipulated in this section. In addition this section should state that LWG will be notified before the Round 2 archived data are purged by the laboratories.

Section A9.2 - Laboratory Documentation: In paragraph 2, change the word "comparable" to "equivalent". Replace the bulleted list of data deliverables with the following:

- A cover letter which lists the LWG samples numbers, Laboratory sample Numbers, and QC samples.

- Case Narrative - Samples analyzed per suite of parameters, include re-analysis and state why re-analysis had to be performed, administrative and technical problems encountered and corrective actions taken, modifications/deviations from the QAPP specified methods, and if possible, Instrument Operating conditions per suite of parameters and a signed authorization of data release by the Laboratory or Project Manager.

- Chain-of-custody, intra and sub-laboratory sample transfer documentation and cooler receipt forms.

- QC Summaries for each suite which include (as applicable): Form Is for all samples, Surrogate Recovery Summary, Matrix Spike and Matrix Spike Duplicate Summary (if available), Laboratory Control and Laboratory Control Duplicate (if available), Method Blank Summary, Instrument Performance Checks, Internal Standard Areas and Retention Time Shift Summary per analytical sequence, Analytical Sequence and Surrogate Retention Time Shift Summary (for GC analyses)

- Sample, Blanks and QC Samples Data - Target Compound Results Summary (Form I), raw data report which includes sample number, volume injected, date and time of injection, GC column identifier, instrument identifier, analyst ID, lab file identifier, target compounds, retention time or scan number of identified compounds, ion used for quantitation, areas/peak heights, concentration on column, final concentration, reconstructed ion chromatograms (RIC) or chromatograms normalized to the highest non-solvent peak, raw and background subtracted spectra of detected compounds, extracted ion current profile (EICPs) displaying all manual. integrations or edits.

- Initial calibration - Initial Calibration Summary, raw data (as listed above) for each concentration level of standard analyzed. Initial calibration verification

summary and raw data..

- Continuing Calibration Verification - Summary of each continuing calibration results and raw data as listed above.

- Instrument Performance Evaluation Checks - Summary of each checks and raw data.

- Miscellaneous Data: Sample Preparation and copies of instrument logbooks, standard traceability to NIST and preparation logs, sample screening and clean-up documentation, communication logs, corrective action reports (if any) concerning the data package.

Section B1.1 - River Surface and Subsurface Sediment:

The last sentence of the 1st paragraph should be corrected to read "231" rather than "131".

Section B1.2 - Shorebird Area and Beach Sediment: The current project schedule calls for shorebird beach sampling in late July 2004. The shorebird breeding season is in the spring. The text should be revised accordingly.

Section B2.1.3, Surface Water: This section will need to be revised based on comments received on the Surface Water FSP.

Section B3.3 - Archived Samples: Adequate sediment volume must be collected and archived for possible PCB congener or other analyses. Table B2-1 states that 2-8 oz of sediment samples will be archived for a year. This amount may not be sufficient for re-analysis or other future analysis. It is highly recommended that, at a minimum, 32 oz. of sediment samples be archived for a year. In addition, the QAPP should specify who will be responsible for archiving the samples.

Section B4.1. - Laboratory Methods for Sediment Samples: PCB congener analysis should be performed on selected PCB aroclor sediment samples. Samples should be selected for PCB congener analysis in consultation with EPA and following a review of PCB aroclor sediment data and PCB congener and aroclor fish tissue data.

Section B4.1.4 - Chlorinated Dioxins and Furans in Sediment: This section states that up to 50 grams of sample will be used for extraction. The lab SOP, MDLs and MRLs listed for PCDDs/PCDFs (Table A6-2), however, indicate that only 10 grams of sample will be extracted and analyzed. This discrepancy should be clarified.

Section B5.2 - Laboratory Quality Control: Project or site-specific measurement quality objectives (MQOs) or PARCC acceptance limits as listed in Tables A7-1 and A7-2 supercede the laboratory established acceptance limits. Remove the last two sentence in this section and provide

the laboratory with copies of these two Tables for compliance with the project DQOs/MQOs.

Section B6 - Instrument/Equipment Testing, Inspection: Delete second sentence in this paragraph.

Section B7 - Instrument/Equipment Calibration and Frequency: Add the frequency of calibration checks for the field instrument (once every ten samples) Multi Probe YSI 556.

Section C1 - Assessment and Response: Suggested language for first paragraph: Assessments planned for Round 2 sampling include: (1) formal field performance and technical audits performed by the Field QA Manager at least once during each field sampling event; (2) technical system audits (TSAs) of the designated laboratories before the sample collection commence and at least once during any phase of the project; (3) technical system audits of the team's data management systems conducted by Integral's QA Manager at least once during any phase of the project; and (4) routine internal performance and peer reviews of each phase of project tasks throughout the duration of the project. These reviews includes routine laboratory performance audits conducted by the laboratory QA Manager, readiness reviews, etc.

If EPA determines it is necessary, an EPA field staff, hydrogeologist and/or QA Officer will accompany the Field QA Manager on at least one of the sampling event audit and during the high volume ultra clean surface water sample collection. In addition, the EPA QA Officer may accompany the Chemistry QA Manager in the TSAs of local labs before sample collection activities commence. State in the QAPP that if persistent problems are identified with the laboratory's generated data, EPA will have the option to conduct a data audit which may include onsite data system management, evidentiary audits and/or GC/MS, GC-ECD tape audits. The Chemistry QA Manager shall accompany EPA during these audits. Written reports identifying potential problems and recommendations will be prepared by EPA and submitted to LWG for prompt corrective action response.

Section D - Data Validation and Usability: Add this statement to this section "Data verification and validation shall be conducted in accordance with the Guidance on Environmental Data Verification and Validation, EPAQA/G8, Final, November 2002".

Section D1 - Criteria for Data Review, Verification and Validation:

The CLP Functional Guidance for Low Concentration Organic Data Review (EPA 2001c) is not be applicable to this project. The QC requirements of the Statement of Work (SOW) that this document was based differ substantially from the SW846 Method 8000s. This guidance document should not be used as a basis for data qualification for ultra low level analyses. For the same reason, the functional guidelines for PCDDs and PCDFs should not be used for this project. The CLP PCDD/PCDF SOW has different technical acceptance, calculations, reporting and QC requirements when compared with Method 1613B.

Add the following guidance document for ultra low level trace metals validation "Guidance on the Documentation and Evaluation of Trace Metals Data Collected for Clean Water Act Compliance Monitoring (EPA 1995) should be used for data verification and evaluation in addition to the specifications of Method 1669 - Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Level (EPA 1996).

The QAPP should state that the first data package generated by each laboratory for each matrix and each suite of parameters will be submitted to EPA for full data validation. It is imperative that laboratory performance and data quality be assessed, potential problems identified and appropriate corrective actions initiated at the beginning of the project. Based on laboratory performance, deliverables and validation results, EPA will set the percentage of data that will require full data validation (QA2) and/or validation based on QC summaries only (QA1). This EPA QA oversight effort should be stated in this section of the Round 2A QAPP.

Sections D1 and D2 should be combined to simplify the discussions. Suggested language for the combined sections is provided below:

The first level of data verification shall be performed by the laboratory performing the analyses wherein the raw data and calculations are verified with the reported results. An independent third party data verification and validation shall also be performed to assess data quality and usability of all the environmental data generated for this project. Data validation shall be performed using the project-specific MQO specifications (Tables A7-1 and 7-2), the analytical methods, the Region10 PCDD/PCDF Data Validation Standard Operating Procedure and the National Functional Guidelines for Inorganic and Organic Data Review.

The quality of the data generated shall be determined during data validation. Random calculation checks and verification of raw data against the reported results shall be performed. Using the data package submitted by the laboratory, the sample collection and laboratory quality system will be assessed by the validator. Critical QA elements directly and/or indirectly affecting data quality shall be evaluated and appropriate data qualifiers shall be applied to the affected data. Data assessment and application of data qualifiers will be in accordance with the following Guidelines/Guidance, the specified analytical methods and/or the validator's sound professional judgment:

- USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review (EPA 1999)
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (EPA 2002g)
- USEPA Region 10 PCDD/PCDF Data Validation Standard Operating Procedure (EPA 1996)

- USEPA "Guidance on the Documentation and Evaluation of Trace Metals Data Collected for Clean Water Act Compliance Monitoring (EPA 1995)

- USEPA Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Level (EPA most recent version)

Analytical results generated for samples collected using high volume samplers for analysis at EPA Water Quality Criteria levels shall be evaluated in accordance with the specified methods and the validator's professional judgment.

EPA will conduct a full data validation of the first data packages generated by each laboratory for every suite of parameters per matrix. The rest of the data generated shall be validated by LWG. Based on the laboratories' performance, deliverables and validation results, EPA will set the percentage of data that will require full data validation (QA2) and the validation based on QC summaries only (QA1) per suite of parameters per matrix.

Table A6-2 - Analytes, Analytical Concentration Goals and Method Reporting Limits for Sediment Samples: The MRLs for chlorinated dioxins and furans are orders of magnitude higher than agreed to by the LWG in the Round 1 QAPP and several orders of magnitude higher than the ACGs. For example the MRL for 2,3,7,8-TCDD is listed as 0.2 pg/g. The Round 1 QAPP listed the MRL for 2,3,7,8-TCDD as 0.01 pg/g. The ACG is listed as 0.0001 pg/g. The MRLs proposed are not acceptable. Revised MRLs must be provided.

For MDLs/MRLs that are listed as to be determined (TBD) the MDL studies and initial demonstration of capabilities (IDCs) per method per instrument need to be submitted by LWG to EPA no later than the end of first week of June to give EPA sufficient time to initially evaluate the laboratories' capabilities in meeting the data quality objectives of this project.

Comments on Table A6-2 are summarized below:

Parameter	**Lab**	**Method**	**ACG/MDL Comment**
TOCs	CAS Kelso	Plumb 1981	OK - Acceptable
Grain Size	CAS Kelso	PSEP Protocol	OK - Acceptable
Total solids	CAS Kelso	PSEP Protocol	OK - Acceptable
Ammonia	CAS Kelso	Plumb, 1981	OK - Acceptable
Total Sulfides	CAS Kelso	EPA 9030	OK - Acceptable
VOCs	CAS Kelso	5035; 8260	MRL- TBD

TBTs	CAS Kelso	Krone et al 1989	MRL- TBD
PCDDs/PCDFs	CAS Houston	1613B	MDL/MRL - not comparable with Round 1 - ACGs and MRLs (Round 2A is much higher than Round 1)
Herbicides & PCP	STL, Tacoma	8151A	TBD
Cr VI	CAS-Kelso	3060A/ 7196A	OK - Acceptable
Geotech Character	CAS Redding		OK - Acceptable
As, Al, As, Cd, Cr, Pb, Ni, Se, Ag, Zn	CAS Kelso	3050/6020	OK - Acceptable
As		3050/7062	OK - Acceptable
Se		3050/7742	OK - Acceptable
Hg		7471/7471A	OK - Acceptable
GRO	CAS - Kelso	NWTPH-GRO	OK - Acceptable
DRO	CAS - Kelso	NWTPH-Dx	OK - Acceptable
Pesticides	NEA	3545; 3640A; 8081A	TBD
SVOCs	NEA	3545; 3640A; 8270C- ITD	TBD
PCB Aroclors	NEA	3545; 3640A; 8082	TBD

Specific Comments - Appendix B:

Section A6 Task Description: The Hyalella 28-day test and the Chironomous 20-day test should be cited as:

> USEPA 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates, second edition, EPA/600R-99/064,

Washington, DC

ASTM. 2003. Standard test methods for measuring the toxicity of sediment-associated contaminants with freshwater invertebrates (ASTM E1706-00). ASTM annual book of standards volume 11.05, ASTM, West Conshohocken, PA.

Section A7 Quality Objectives and Criteria: Change 10-day to 20-day for C tentans.

Section A9.2, Testing Results: The QAPP states that "average percent survival" and "average biomass" will be presented in the electronic package that summarizes the results. All replicate data, not just averages, should be presented for each bioassay sample.

Section A9.3 - Data Reduction: The QAPP states on page 8 that a "data reduction" will take place. Raw data which contains the replicate information will be "reduced" to average survival and biomass, and that the QA/QC and the validation including the QA report (by Dinnel Marin Resources) will take place on this reduced set. Validation and QA/QC should take place on the raw (replicate) data set in addition to the reduced data set.

Section B1 - Experimental Design: The QAPP should present interpretative criteria for the bioassay tests. Initial "hit/no hit" criteria based on a statistical difference from negative control consistent with ASTM and EPA methodology should be included.

Tables B1-1 and B1-2 - Summary of Test Conditions:

The main text of Appendix B states that the overlying (test) water will be "Moderately hard synthetic water." In Attachments #2 and #3 the specifications for the overlying water are provided as hardness of 80-100 mg/L of $CaCO^3$ and alkalinity of 60-70 mg/L as $CaCO^3$. The hardness the synthetic water is quite a bit higher than that of the Willamette River (100 mg/L versus 30 mg/L, respectively) which may alter bioavailability and toxicity. As a result, moderately hard synthetic water may not amenable to testing with the selected test organisms. ASTM and EPA recommend using natural water for testing with these organisms. It appears the lab has access to natural water (Attachment 1, Page 29), which may be evaluated for use in these tests. The water hardness during testing should be as close a possible to the system we are evaluating.

Reconstituted water should not be used to conduct 28-day H. azteca sediment test. The same overlying water should be tested in the amphipod and midge tests (Table B2-2)

Water quality - include sulfide in Table B1-1 and B2-2.

Section B4 - Toxicity Test Method Requirements: Method and acceptability criteria from ASTM,

2003 should also be followed (ASTM, Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates).

Section B5, Laboratory Quality Assurance/Quality Control: It is stated that the negative control sediment will be collected from Beaver Creek in Lincoln City. The physical and chemical characteristics of this sediment should be described, so the applicability as a control sediment to the Willamette River can be evaluated. Also, indicate if this sediment has been used successfully in the past to conduct the midge and amphipod tests. ASTM and EPA recommend conducting 5 tests with a negative control before it is used to assess test sediments.

Ammonia and sulfide should also be measured in pore water at the beginning and end of the exposure and reported; pH and dissolved oxygen of the pore water should also be monitored.

Section D2, Date Review and Validation: This section states "all data will be accessible, but only validated data will be released for general use. The "validated data" will only be the "reduced set" reviewed by Dinnel Marine Resources. The complete raw data set should be available for general use, including the replicate information.

Section E References: See comment for A6 above.

Attachment #2, Test Protocol for the 28-day *Hyalella* test

Section 10 - Reporting: Replicate data should be reported, not just the means and standard deviations for each test sediment.

Section 5.4 Test Organisms, Acclimation and Pretest Observation: acclimation of test organisms to the low hardness test water may be necessary.

Attachment #3, Test Protocol for 10-day *Chironomus* Test: At this time, EPA has directed the LWG to perform the 20-day Chironomus Test. The QAPP must include the appropriate test protocol.

Test organism section: "excessive" mortality is not defined in the QAPP. ASTM and EPA recommend <20% mortality for 48 hours before the start of a test.

Effect criterion: delete "acute" from reference to mortality as criterion.

Test conditions summary: see previous comments on reconstituted water

Attachment #4, Examples of electronic data submittal for the toxicity tests: The included sheets show replicate survival and mortality - not just average results and summary statistics (e.g., means and standard deviations). Please confirm that this information will be available for "general use."

Specific Comments - Laboratory QA Manuals:

PCB and Organochlorine Pesticide Analysis - SW846 Methods 8082 and 8081-
Laboratory - Northeast Analytical Inc. (NEA)

1. For point of reference, add surrogates (TCX and DCB) to all Aroclor standards.

2. Submit GPC calibration verifications performed at least once every 7 days and corresponding raw data.

3. Submit Florisil cartridge performance checks performed for every florisil lot using the fractionation techniques that will be employed for sample clean-up for this project and the corresponding raw data.

4. The standards and samples chromatograms must display the peaks chosen for identification of each analyte at greater than 30% and <100% of full scale.

5. If a chromatogram is re-plotted electronically, both the initial chromatogram and the re-plotted chromatogram must be submitted with the data package.

6. If an Aroclor is detected in the sample, the Aroclor detected must be analyzed in the continuing calibration within 72 hours of detection.

7. If more than one Aroclor is observed in a sample, the laboratory must choose different peaks to quantitate each analyte. A peak common to both analytes present in the sample must not be used to quantitate either compound.

8. Detected pesticides and Aroclors in the sample using dual column GC-ECD must have the identification confirmed by GC/MS if the concentration is sufficient for that purpose. Raw and background subtracted spectra of three characteristic peaks of the Aroclor and pesticide peak identified should be submitted with the package. GC/MS confirmations can be performed by either reverse TIC search using the SVOC run or the pesticide and/or Aroclor extract.

9. Sample extracts will not be disposed by the laboratory without the approval of LWG.

10. Dionex Accelerated Extraction usually affects surrogate recoveries. Include the surrogates in the Method Detection Limit (MDL) studies. MDLs and IDCs must be submitted to EPA as soon as possible for evaluation and determination of laboratory capabilities.

11. Because the non-detects will be reported at the MDL, a standard at the MDL level must be analyzed at least once at the beginning of each analytical sequence.

12. For ultra low analyses, it is highly recommended that storage holding blank (refrigerator

blanks) be extracted and analyzed with the samples. A holding blank is a volume of clean reference matrix stored with the samples while waiting extraction. Holding blanks determines cross- contamination during storage.

Semi-volatile Organic Compound (SVOC) Analysis by SW846 Method 8270C
Laboratory - NEA

1. It should be noted that the reporting limits and the MDLs submitted by lab will be based on the analysis of clean extracts. Ion trap mass specs are very good for clean sediment or water samples. If the samples are heavily contaminated, however, mass ion resolutions are greatly affected and thus the auto gain function of the instrument is triggered to do ionization time modulation. Modulation means that the mass ionization time will be much less than normal (depending on the amount of material ionizing in the trap), baseline and noise levels increase which eventually lead to dynamic changes in the detection limits. Instead of collecting certain amount of ions, lesser target compound ions are collected due to the presence of other organic material in the extract, the outcome will be higher detection limits and low biased results. The laboratory should compensate for these detection limit changes. It is recommended that for heavily contaminated samples (as would be shown by the chromatogram), the laboratory must elevate the detection limits at levels $\geq$ the baseline and /or noise levels in the samples and the concentrations of the detected compounds should be flagged as estimated.

2. Because the non-detects will be reported at the MDL, a standard at the MDL level must be analyzed at least once at the beginning of each analytical sequence.

3. For ultra low analyses, it is highly recommended that storage holding blank (refrigerator blanks) be extracted and analyzed with the samples. A holding blank is a volume of clean reference matrix stored with the samples while waiting extraction. Holding blanks determines cross- contamination during storage. Holding blanks can be analyzed outside the 12- hour QC period.

4. Dionex Accelerated Extraction usually affects surrogate recoveries. Include the surrogates in the Method Detection Limit (MDL) studies.

PCDDs/PCDFs - Laboratory Columbia Analytical Services, Houston TX

1. The lab SOP, MDLs and MRLs listed for PCDDs/PCDFs indicate that only 10 grams of sample will be extracted and analyzed. The MDLs and MRLs that will be reported by the laboratory are not consistent and are much higher than Round 1. During Round 1 50 grams of sediment samples were extracted and analyzed by Axys lab. It is highly recommended that LWG require the lab to extract more samples and make the necessary adjustments with the initial calibration range to get a much lower MDL and MRL than what are currently listed in the Round 2A QAPP.